

# Novel Carbon Nanotube-Based Nanostructures for High-Temperature Gas Sensing

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## OBJECTIVES

The primary objective of this research is to examine the feasibility of using vertically aligned multi-wall carbon nanotubes (MWCNTs) as a high temperature sensor material for fossil energy systems where reducing atmospheres are present. The research will be pursued in three main areas: 1) study the growth mechanisms of MWCNTs using the flame synthesis technique and modification of the nanotemplate to improve the quality of the nanotubes for use in a gas sensing platform, 2) transform the modified vertically aligned CNTs into a capacitive type hydrogen sensor prototype to assess feasibility for high temperature applications, and 3) pursue theoretical modeling and numerical simulation of nanostructures, hydrogen gas sensors, and the flame synthesis for large-area nanotube growth.

## ACCOMPLISHMENTS TO DATE

### Nanostructure Modification and Characterization

Vertically aligned carbon nanotubes are currently fabricated using anodic aluminum oxide (AAO) nanotemplate. The pore size or diameter of nanotubes is varied using different voltages, electrolyte, and temperature. The pore wall and barrier layer are variables in modifying the nanotemplate through control of the acidic solution strength. The nanostructure of the template has been studied in detail using SEM. Fig. 1 shows the typical SEM image of an AAO template and carbon nanotubes grown from the template.

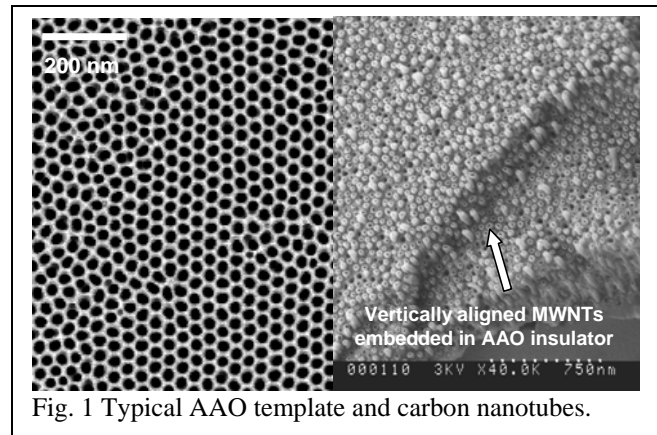


Fig. 1 Typical AAO template and carbon nanotubes.

### Nanotube Growth

Growth of the carbon nanotubes is carried out using flame synthesis technique. The apparatus consists of two concentric tubes. The inner tube, made of stainless steel, is 1.1 cm in diameter. The outer tube is made of copper and has a diameter of 5 cm. A steady and stable laminar diffusion flame with a visible flame height of 33 mm and 20 mm is established on the burner port. The experimental results show that the position in the flame suitable for synthesis of CNTs is about 4 mm and 9mm above the burner exit for ethylene and methane. The AAO template (10 mm×5 mm) was inserted into the flame perpendicular to the gas flow direction and kept in place for 15 min. The pyrolyzed gaseous products were impinged on the template. With the heat generated at flame sheet, the gas environment near the template was suitable for CNT synthesis with a temperature of 1200–1500°C. Visual inspections showed a layer of smooth and shiny black material on Samples. To observe the CNTs inside the AAO pores under SEM, the samples

were dipped in the chromic and phosphoric acid mixture to remove AAO templates. In the past, we use acetylene flame in the presence of cobalt catalyst to grow CNTs. After CNT growth, we found thick amorphous carbon film formed on the surface of AAO templates where CNT grew. This amorphous carbon film is very hard to remove to get a clean surface, which is necessary to fabricate CNT sensors or other micro/nano devices. Various attempts, such as time, temperature, and atmosphere and catalyst size of CNT growth, were made to solve this problem. Finally a clean surface with perfect CNTs was obtained by changing ethylene to methane and correspondingly adjusting some flame parameters. From Fig. 2, it can be seen that much cleaner surface is obtained using methane, which is crucial for the following fabrication steps.

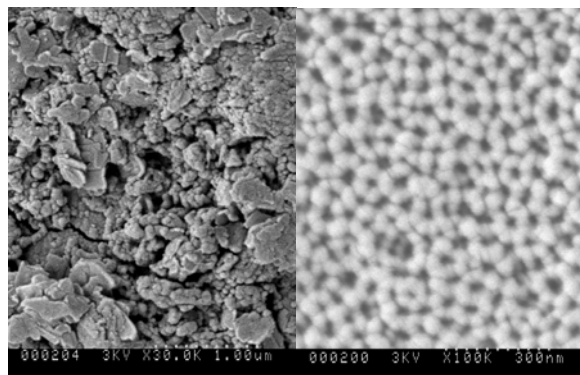


Fig. 2 SEM pictures of sample surfaces after CNT growth in ethylene (Left) and methane (Right).

### Design of Hydrogen Sensors

Two types of capacitive sensors structures have been designed. One structure involves the direct deposition of the top metal electrode on the aluminum oxide embedded with nanotubes. The other structure has an insulator layer between the metal and the aluminum oxide embedded with nanotubes. The sensors' response to a reducing gas (hydrogen) environment at room temperature will be tested.

### FUTURE WORK

#### Establishment of the High-Temperature Test Setup

The quartz tube furnace will be used for construction of a setup for high temperature gas sensing measurement. The concentration of gases will be monitored by flow meters and temperature will be monitored by thermocouples. The final set up will consider the impact of a reducing environment under elevated or high temperatures. The use of gas mixture containing hydrogen at elevated temperature will be reviewed for safety.

#### Hydrogen Sensor Modeling

The sensors will be modeled on considering the gas molecule diffusion into the nanotubes and the electrical models will be constructed on considering the 3-D structure. This work will lead to a better understanding of the types of nanotubes and sensor structures that are preferred for sensing in a high temperature atmosphere.

#### Fabrication of Hydrogen Sensors for Use in High Temperature

Capacitive sensors will be fabricated based on the nanotube-based nanostructures, which are able to withstand high temperature up to 1000°C. Special electrodes and connection wires being able to withstand high temperature will be used.

### LIST OF STUDENTS SUPPORTED

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